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METHOD FOR DETERMINING THE CHLORIDE CONTENT IN ORGANIC ADDITIVES FOR PORTLAND CEMENT CONCRETE

CAUTION: Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read "**SAFETY AND HEALTH**" in Section F of this method. It is the responsibility of the user of this method to consult and use departmental safety and health practices and determine the applicability of regulatory limitations before any testing is performed.

A. SCOPE

This method includes two procedures, potentiometric and gravimetric, used to determine the chloride content of organic compounds used as additives in portland cement concrete. These compounds are usually lignosulfonate derivatives and are received in the form of liquids or dry powders.

Ordinarily, the materials should contain less than 1 % chloride. The gravimetric method is used when the potentiometric method gives chloride greater than 0.8 %, expressed as chloride.

PART I: POTENTIOMETRIC

B. REAGENTS AND SPECIAL APPARATUS

1. Unless otherwise indicated, all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society.

2. A pH meter, with a mV-scale, low drain, sensitive to 1 mV

3. Double junction reference electrode

4. Chloride specific ion electrode

5. Magnetic stirring bar and stirring plate

6. Electrode assembly: Refer to the literature accompanying electrodes for the proper filling solutions, maintenance and operating procedures.

C. TEST PROCEDURE

1. Weigh 10.0 g of the material into a 400-mL beaker.

2. Add 200 mL of deionized water.

3. Add 20 mL of concentrated nitric acid and stir.

4. Carefully introduce a magnetic stirring bar.

5. Center the beater on the stirring plate, and immerse the electrodes into the sample. Make certain that the level of the outer solution of the double junction reference electrode is at least 25 mm above the level of the sample solution in the beaker.

6. Stir at a moderately fast rate, but do not allow the vortex to go below the ends of the electrodes.

7. Turn the pH meter to the "on" position and allow it to stabilize, if necessary.

8. Titrate with 0.1 N silver nitrate. As the end point is approached, the change in the millivolt reading will significantly increase, at which time, add the silver nitrate in small equal volume increments (e.g., 4 drops). Record potential [E] in mV against titration volume [Vol] in mV after each increment. Beyond the end point, the meter reading will decrease appreciably.

9. Determine the end point by the following method:

   Plot the curve of E (potential) against Vol (volume). Find the point of inflection and read the volume at this point.

D. CALCULATION

% Cl = (mL of $AgNO_3$ X normality of $AgNO_3$ X 3.55) / mass of the sample

PART II: AUTOMATIC TITRATOR

B. REAGENTS AND MATERIALS

1. Standard silver nitrate titrant, 0.0141 N. Dissolve 2.395 g of $AgNO_3$ in deionized water and dilute to 1000 mL.

2. Nitric acid, concentrated HNO_3

   Unless otherwise indicated, all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society.

C. TEST PROCEDURE

1. Weigh 10.0 g of the material into a 400-mL beaker.

2. Add 200 mL of deionized water.

3. Add 20 mL of concentrated HNO_3.

4. Set the titrator instrument to the desired parameters by following the manufacturers instructions.

5. Titrate the sample with silver nitrate solution.

D. CALCULATION

$$\% \text{ Cl} = \frac{(A-B) \times N \times 3.55}{\text{(mass of the sample)}}$$

where:

A = mL of $AgNO_3$
B = mL of the blank
N = normality of $AgNO_3$

E. REPORTING RESULTS

Report the results on an appropriate test form.

F. SAFETY AND HEALTH

This method may involve hazardous materials, operations, and equipment. This method does not purport to address all the safety problems associated with its use. It is the responsibility of whoever uses this method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Prior to handling, testing or disposing of any of waste materials, testers are required to read: Part A (Section 5.0), Part B (Sections: 5.0, 6.0, 10.0 and 12.0) and Part C (Section 1.0) of Caltrans Laboratory Safety Manual. These sections pertain to requirements for general safety principles, standard operating procedures, protective apparel, disposal of materials and how to handle spills, accidents, emergencies, etc. Users of this method do so at their own risk.

REFERENCES:

None

End of Text (California Test 415 contains 2 pages)